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### TRANSLATION

BERMAN PATENT 28 32 679 ASSIGNEE: Henkel KGAA INVENTOR: Hans Andree FUBLICATION DATE: 2/13/79

#### COLD WASH PROCESS

#### CLAIMS

- 1. Low temperature machine washing process for textiles using from 2 to 12 g/1 of a laundry product containing surfactants and builder salts as well as optionally bleaching agents and other laundry product components, distinguished by the fact that the load of textiles is washed with the use of a laundry product containing
- A) 3 to 15 wt% of at least one nonionic surfactant, which has a hydrocarbon group of 10 to 20 carbon atoms and 3 to 20 ethylene glycol ether groups,
- B) 0 to 20 wt% of at least one anionic surfactant from the group of sulfonates, sulfates and soaps, having a hydrocarbon group of 10 to 24 carbon atoms,
- C) 20 to 50 wt% of at least one water soluble or water insoluble builder salt which binds alkaline earth metal ions.
- D) 0.3 to 3 wt% of at least one polyamide containing hydroxyalkyl or polyalkylene glycol ether groups, having a cloud point in water determined according to DIN 53719 above 20°C and/or at least one compound of the following formulas

$$R^{1} - CH_{2} - CO - \left[ \begin{array}{c} N - (CH_{2}) \\ 1 \\ Y \end{array} \right]_{P} - N \left( \begin{array}{c} (CH_{2}CH_{2}O) \\ M \end{array} \right)_{m} - H$$
(1)

$$R^{1} - CH - CH - \frac{C}{Y} - \frac{N}{Y} - (CH_{2})_{q} - \frac{(CH_{2}CH_{2}O)_{m} - H}{Y}$$
(II)

$$R^{1} - CH - CH - R^{2}$$
 $X = HN - \left[ (CH_{2})_{q} - HH \right]_{p} - COCH_{2}OCH_{2}CH_{2}OH$ 
(III)

$$R^{1} - CH - CH - R^{2}$$

$$O_{CO} = (CH_{2})_{q} - H(CH_{3})_{2}$$
(IV)

in which  $R^1$  represents an alkyl or alkenyl group of 8 to 20 carbon atoms,  $R^2$  is hydrogen or an alkyl group of 1 to 16 carbon atoms, and  $R^1 + R^2$  contain 8 to 20 carbon atoms altogether, X = H, DH or the group  $\{CH_2CH_2D\}_{N} - H$ , Y the group  $\{CH_2CH_2D\}_{N} - H$ , M = 1 to 3, and P = 8 or 1 and P = 2 or 3,

- E) 0.05 to 1 wtx of a suds suppressing dimethyl polysiloxane,
- F) ® to 48 wt% of other laundry product components,

in a wash liquor ratio (kg textile load to liter wash liquor) of 1 g 3 to 1 g 30 for a time period of 20 to 75 minutes (=  $t^1$ ) at a temperature of 15 to 3500 (=  $t^2$ ), and the sum of the non-dimensional figures  $t^1$  +  $t^2$  = 35 to 50, and the wash load is mechanically agitated during at least a third of the washing time, periodically interrupted by periods of rest.

- 2. Process according to Claim 1, distinguished by the fact that the use concentration of the laundry product is 4 to  $10^\circ$  g/l.
- 3. Process according to Claims 1 and 2, distinguished by the fact that the wash liquor ratio is 1 : 4 to 1 : 20.
- 4. Process according to Claims 1 to 3, distinguished by the fact that the wash period is 30 to 60 minutes, the temperature is 20 to 30°C and the sum of  $t^1$  +  $t^2$  = 50 to 80.
- 5. Process according to Claims 1 to 4, distinguished by the fact that the wash load is mechanically agitated during half of the washing time, periodcially interrupted by rest periods.
- 6. Process according to Claims 1 to 3, distinguished by the fact that the washing is done using a laundry product in which the component (A) consist of a mixture of ethoxylated primary alcohols which have 12 to 18 C atoms, with a low ethoxylated portion having 3 to 6 glycol ether groups and a highly ethoxylated portion having 7 to 16 glycol ether groups and the weight ratio of low to high ethoxylated portions being 3 : 1 to 1 : 3.
- 7. Process according to Claims 1 to 6, distinguished by the fact that the washing is done using a laundry product in which the component (B) is contained in a proportion of 1 to 15 wt% and consists of an alkylbenzene sulfonate having 8 to 13 C atoms in the alkyl chain and/or an alkane sulfonate having 12 to 18 C atoms and/or a soap having 12 to 18 carbon atoms.
- B. Process according to Claims 1 to 7, distinguished by the fact that the washing is done using a laundry product in which the component (C) consists of an alkali metal polyphosphate and/or the alkali metal salts of an aminopolycarboxylic acid or polyphosphonic acid and/or a water containing sodium aluminosilicate of the formula  $\{Na_2D\}_X$ .  $\{Na_2D\}_Y$  with  $X = \emptyset$ .  $\{Na_2D\}_Y$  and  $Y = \emptyset$ .  $\{Na_2D\}_Y$  by  $\{Na_2D\}_Y$  and  $\{Na_2D\}_Y$  by  $\{Na_2D\}_Y$  by

9. Process according to Claims 1 to 8, distinguished by the fact that the washing is done using a laundry product in which the weight ratio of components (A+B) to component D is 30:1 to 2:1.

## COLD WASHING PROCESS

The subject of the present invention is a cold water machine washing process with controlled sudsing.

Except for special laundering procedures, which for example are used for delicate synthetic, silk and wool textiles, and in which highly dilute wash liquors of at most less than 2 g of laundry product per liter, a load ratio of at least 25 liters of wash solution per kg of wash load at temperatures below 40°C and the least possible mechanical agitation of the load are used, for the normal household wash temperatues of 50 to 95° are required in order to obtain satisfactory washing results. Since in the domestic washing machines generally in use today the heating of on the average 20 to 40 liters of wash solution is necessary and the increased wash temperature is generally maintained for 10 to 15 minutes, the energy consumption is considerable.

There has not been a lack of studies directed toward saving this energy by lowering the wash temperature. Thus before the introduction of modern wash methods and machines it was usual to soak the wash for a long time — generally overnight — in a dilute wash solution. In this way, however, only a very small amount of soil is dissolved. The same is true for the "washing cycle" in domestic washing machines which precedes the so-called clear of main wash cycle, and in which the wash liquor is heated slightly or not at all. Stubborn, especially fatty soils are not raemoved by this pretreatment.

In DT-AS 12 62 207 a cold washing process is described using optical bleach and strong, especially active chlorine containing bleaching agent, in which the wash load is introduced into the wash liquor at room temperature, and is mechanically agitated for a particular time at the beginning and at the end of the washing process. In order to obtain satisfactory washing results, however, the laundry must remain in the wash liquor for at least 10 hours, which is considered to be unreasonable by many users.

The present invention here offers a significant improvement. It concerns a machine washing process for textiles at low temperature using from 2 to 12 g/l of a laundry product containing surfactants and builder salts, as well as optionally bleaching agents and other laundry product components, which is distinguished by the fact that the textile load is washed with the use of a laundry product containing

A) 3 to 15 wt% of at least one nonionic surfactant which has a hydrocarbon group of 10 to 20 carbon atoms and 3 to 20 ethvlene

B)  $\Theta$  to  $2\Theta$  wtx of at least one anionic surfactant from the group of sulfonates, sulfates and soaps, whose hydrocarbon chain contains  $1\Theta$  to 24 carbon atoms,

C) 20 to 60 wt% of at least one water soluble or water insoluble builder salt which binds alkaline earth metal ions,

D) 8.3 to 3 wt% of at least one polyamide which contains hydroxyalkyl or polyalkylene glycol ether groups, whose cloud point in water - determined according to DIN 33917 - is above 2000 and/or at least one compound of the following formulas

$$R^{9} - CH_{2}-CO - \left[\begin{matrix} H - (CH_{2}) \\ Q \\ \end{matrix}\right]_{p}^{-H} \left(\begin{matrix} CH_{2}CH_{2}O \end{matrix}\right)_{m}^{-H}$$
 (1)

$$\mathbb{R}^{1} - CH - CH - \left[\frac{1}{N} - (CH_{2})^{2}\right]^{-N} < \frac{(CH_{2}CH_{2}O)^{m}-H}{2}$$
 (XI)

$$R^{9} = CH - CH - R^{2}$$

$$0 \qquad N = (CH_{2})_{q} - N(CH_{3})_{2}$$
(IV)

in which  $\mathbb{R}^1$  represents an alkyl or alkenyl group having 8 to 20 carbon atoms,  $\mathbb{R}^2$  is hydrogen or an alkyl group having 1 to 16 carbon atoms,  $\mathbb{X} = \mathbb{H}_1$  OH or the group (CH2CH2D) $_{\mathrm{N}}$ -H, Y is the group H or (CH2CH2D) $_{\mathrm{N}}$ -H, m = 1 to 3, n = 8 to 3, and p = 8 or 1 and q = 2 or 3,

E) 8.85 to 1 xtx of a suds suppressing dimethyl polysiloxane,

F) B to 48 xt% of other laundry product components,

at a wash liquor ratio (kg textile load to liter wash liquor) of 1  $_{1}$  3 to 1  $_{1}$  3% for a time period of 2% to 7% minutes (=  $t^{1}$ ) at a temperature of 1% to 3% (=  $t^{2}$ ), the sum of the non-dimensional figures  $t^{1}$   $\Leftrightarrow$   $t^{2}$  = 3% to 9% and the wash load is mechanically agitated during a third of the wash time, interrupted by periods of rest.

The use concentration of the laundy product is preferably 4 to 10 g/l, with a recommended wash liquor ratio of 1 s 4 to 1 s 20. The washing time under these conditions is preferably 30 to 60

+ to = 50 to 80. The wash load is preferably mechanically agaitated during half of the washing time at periodic intervals. This agitation can be done by tumbling the wash in a rotating wash drum or by a rotating vane agitator in stationary wash drums, in which optionally the direction of movement is periodically reversed. The speed of revolution of the moving device of 8.3 to 3 revolutions per sec. is considered sufficient. These figures on speed of revolution do not apply for the spin cycle after the wash liquor has drained.

The nonionic surfactants contained in the wash liquor (Component A) are those from the group of polyglycol ether derivatives having 3 to 30 gylcol ether groups and 10 to 20 carbon atoms in the aliphatic group or 5 to 14 carbons atoms in the alkyl chain of an alkylaromatic group. Especially suitable are mixtures of such polyglycol ether derivatives in which at least one compound has 3 to 5 glycol ether groups and at least one compound has 7 to 15 glycol ether groups in a weight ratio of 1 : 5 to 5 : 1. Polyglycol ether derivatives of primary alcohols of 12 to 18 carbon atoms having a straight chain or branched at the 2-position and of alkylphenols with straight chain alkyl groups of 8 to 12 carbon atoms are preferably used. Dther nonionic washing components which can be used are glycol ether derivatives of fatty acids, fatty acid amides, primary or secondary amines, vicinal diolens, thioalcohols and sulfamides.

Buitable anionic surfactants (Component B) are especially alkali soaps of fatty acids of natural or synthetic origin, e.g. the sodium salts of coconut, palm kernel or tallow fatty acids. Others are surfactants of the sulfonate or sulfate type, especially alkane sulfonates and alkylbenzene sulfonates, such as n-dodecylbenzene sulfonate. Further useable are olefin sulfonates and alpha—sulfo fatty acid esters, primary and secondary alkyl sulfates as well as the sulfates of ethoxylated or propoxylated high molecular weight alcohols, the sulfated partial ethers and partial esters of polyvalent alcohols, such as the alkali salts of monoalkyl ether and the monofatty acid ester of glycerin monosulfuric acid ester and 1,2-dioxypropane sulfonic acid. Sulfates of ethoxylated or propoxylated fatty acid amides and alkylphanols as well as fatty acid taurides and fatty acid isothionates can also be used.

The anionic surfactants are preferably present in the form of the sodium salts, possibly also the potassium salts and the salts of organic bases, such as mono-, di- or triethanolamine. If the anionic compounds mentioned contain an aliphatic hydrocarbon group, this should preferably be straight chain and have 12 to 18 carbon atoms. In the compounds with an aryl-aliphatic hydrocarbon group the preferably unbranched alkyl chains contain on the average 8 to 13 carbon atoms.

The proportion of Component (B) is preferably 1 to 13 wtx.

Builder materials which bind alkaline earth metal ions (Component

which can be present in a mixture with its products of hydrolysis, the mono- and diphosphates, as well as higher condensed phosphates, e.g. the tetraphosphates.

The phosphate polymers can also be replaced wholly or partly by phosphate free sequestering agents. Among these are the alkali salts of amino polycarboxylic acids, especially nitrilotriacetic acid and ethelenediamine tetraacetic acid as well as their higher homologs, further the alkali salts of amino and hydroxyalkyl polyphosphonic acids, for example aminotri-(methylenephosphonic acid), 1-hydroxyethane-1,1-diphosphonic acid and salts of the higher homologs of these polyphosphonic acids.

Buitable water insoluble builders are especially water containing alkali aluminum silicates, which may contain bound water in which the alkali metal can be exchanged by calcium or magnesium. Among these are especially finely crystalline aluminum silicates of the formula  $\{Na_2D\}_X$ .  $\{RiD_2\}_Y$ , where x is a number from 0.7 to 1.3 and y is a number from 1.3 to 4. Also mixtures of the above water soluble and water insoluble builders and complexing agents can be used.

Nitrogen and phosphorus free polycarboxylic acids, which complex salts with calcium ions, can also be used, and polymers containing carboxyl groups are among these, and can be used as builders. Suitable are citric acid, tartaric acid, benzenehexacarboxylic acid and tetrahydrofuran tetracarboxylic acid. Polycarboxylic acids containing carboxymethyl ether groups can be used, such as 2,2°-oxydisuccinic acid, polyvalent alcohols or hydroxycarboxylic acids partly or completely etherified with for example triscarboxymethyl glycerin, glycolic acid, biscarboxymethyl glyceric acid and carboxymethylated or oxidized polysaccharides. Further suitable are the carboxylic acid polymers having a molecular weight of at least 350 in the form of the water soluble sodium or potassium salts, such as polyacrylic acid, polymethacrylic acid, poly-alpha-hydroxyacrylic acid, polymaleic acid, polyitaconic acid, polymesaconic polybutene tricarboxylic acid as well as the copolymers of the corresponding monomeric carboxylic acids with one another or with ethylenically unsaturated compounds such as ethylene, propylene, isobutylene, vinyl methyl ether or furan.

Component (D), which is present preferably in proportions from 8.5 to 2 wt%, consists of amine or amide derivatives dispersed in water, most of which contain hydroxyalkyl groups. Compounds of Formula (1) can consist of alkoxylated acylmonoethanolamides or - diethanolamides or ethoxylated acylalkylenediamines, which are derived from fat groups of natural or synthetic origin.

Compounds of Formula II can be obtained by reacting long chain epoxyalkanes with mono- or diethanolamine or hydroxyalkylated diamines. Typical representatives of Formula II are for example the following reaction products (cloud points measured in aqueous butyl diglycol according to DIN 53917):

- a) The reaction product of an internal  $E_{11}$ - $E_{14}$  epoxyalkane and diethanolamine; cloud point (  $8^{\circ}E$ .
- b) The reaction product of an internal  $C_{II}$ - $C_{I4}$  epoxyalkane and monoethanolamine; cloud point 34°C.
- c) The reaction product of an internal  $E_{11}$ - $E_{14}$  epoxyalkane and bis-hydroxyethoxy ethylamine; cloud point 44°C.
- d) The reaction product of an internal  $\mathbb{C}_{11}$ - $\mathbb{C}_{14}$  epoxyalkane and diethanolamine, additionally ethoxylated with one mole of ethylene oxide; cloud point 32°C.
- e) The reaction product of an internal  $\mathbb{C}_{11}$ - $\mathbb{C}_{14}$  epoxyalkane and diethanolamine, additionally ethoxylated with 2 mols of ethylene oxide; cloud point 45°C.
- f) The reaction product of an internal  $E_{11}$ - $E_{14}$  epoxyalkane and ethelenediamine, additionally reacted with 4 mols of ethylene oxide; cloud point 72.5°C.
- g) The reaction product of alpha-epoxyoctane and ethylenediamine; cloud point 15°C.

Additional suitable compounds of this class are given in DE-PA 2839/77 (DE 3537).

Compounds of Formula (111) are substituted beta-hydroxyethoxy acetamides, as described in DT-DS 27 29 209.7 (D 5407). They can be derived from terminal or internal monoamines or from reaction products of long chain epoxyalkanes. Suitable compounds of this class are e.g.

N-octyl-beta-hydroxyethoxy acetamide
N-decyl-beta-hydroxyethoxy acetamide
N-tetradecyl-beta-hydroxyethoxy acetamide
N-coconut alkyl-beta-hydroxyethoxy acetamide
N-tallow alkyl-beta-hydroxyethoxy acetamide
N-(1+)-C10-C13-alkyl-beta-hydroxyethoxy acetamide
N-(1-C14-C15-alkyl)-beta-hydroxyethoxy acetamide
N-(1-C17-C20-alkyl)-beta-hydroxyethoxy acetamide
N-(2-hexyldecyl)-beta-hydroxyethoxy acetamide
N-(3-dodecylaminopropyl)-beta-hydroxyethoxy acetamide
N-(2-dodecylaminoethyl)-beta-hydroxyethoxy acetamide
N-(2-hydroxy-i-C11-C14-alkyl)-beta-hydroxyethoxy acetamide
N-(2-hydroxy-i-C15-C18-alkyl)-beta-hydroxyethoxy acetamide

Dxazolidones of Formula (1V) can be obtained from long chain epoxyalkanes by reaction with urea and N,N-dimethylalkylene diamines. An example for this is the oxazolidone from terminal

or internal C12-C18 epoxide, urea and dimethyl propylenediamine.

The components of the polyamides containing glycol ether groups given under Component (D) are those in which first during their

mixtures are polycondensed, then the polyamide is reacted with ethylene oxide. Such polymers are given in DT-DS 27 28 243.9 (D 5372). They are also such polyamides whose acid component consists of polyalkeneoxy-bis-(alkylamines) of the formula

in which 2 represents an ethylene, propylene or trimethylene group, R<sup>3</sup> is an ethylene or propylene group and m is the numbers 1 to 30, preferably 2 to 10. These polycondensation products are the subject of DE-PS 3164/77 (DE 5503).

Examples of alkoxylated polyamides are:

- a) The reaction product of the polyamide from hexamethylenediamine, adipic acid and epsilon-caprolactam (Ultramid 6A) with ethylene oxide, in a weight ratio of 1: 1.37. Cloud point >  $100^{\circ}$ C.
- b) The reaction product of 56.3 g of the polyamide Ultramid 6A and 6B g of ethylene oxide; cloud point > 100°C.
- c). The reaction product of 56.3 g of the polyamide Ultramid 6A first with 48 g of ethylene oxide and then with 87 g of propylene oxide; weight ratio of polyamide z epoxide 1 z 2.4; cloud point 37 39°C.
- d) The reaction product of 56.3 g of the polyamide Ultramid 6A with first 385 g of ethylene oxide and then 103 g of propylene oxide; weight ratio of polyamide: epoxide 1 z 8.6; cloud point >100°C.
- e) The reaction product of 56.3 g of the polyamide Ultramid 6A with first 77 g of ethylene oxide and then 103 g. of propylene oxide; cloud point  $47-49^{\circ}C$ .
- f) The reaction product of the polyamide from hexamethylene diamine, adipic acid, epsilon-caprolactam and 4,4°-diamino-1,1°-dicyclohexyl-methane (Ultramid 1D) with ethylene oxide in a weight ratio of 1  $\pm$  1.56; cloud point >180°D.
- g) The reaction product of the polyamide from hexamethylene diamine and adipic acid (Ultramid R3) with ethylene oxide in a weight ratio of 1 s 4.1s cloud point >  $180^{\circ}$ C.
- h) The reaction product of the polyamide from epsilon-caprolactam (Ultramide B3) with ethylene oxide in a weight ratio of 1 s 4.4s cloud point > 180°C.
- i) The reaction product of a poly-alpha-caprolactam and ethylene oxide, with an ethylene oxide content of ca. 77 wt%; cloud point > 1000 {commercial product Lurotex ACS of BASE Co. >.
- k) The reaction product of the polyamide from hexamethylene

weight ratio of 1 : 5.3. Cloud point > 100°C.

Examples for polyamides from dicarboxylic acids and polyglycol diamines are:

- 1) The reaction product of 1 mol of succinic acid and 1 mol of the polyglycol diamine prepared from a polypropylene glycol of molecular weight 420 and with a degree of hydration of ca. 99%; cloud point of the product in water by DIN 53917; 30-32°C.
- m) The reaction product of 1 mol of sebacic acid and ca. 2.2 mol of the polyglycol diamine, prepared from a polyethylene glycol of molecular weight 200 and a degree of hydration of ca. 45%; cloud point of the product in water by DIN 53917: 34-35°C.
- n) The reaction product of 1 mol of adipic acid and 1 mol of the polyglycol diamine prepared from a polypropylene glycol of molecular weigh 420 and with a degree of hydration of ca. 99%; cloud point of the product in water by DIN 53917: 28-30°C.

Dbviously, with the above polymers the various dicarboxylic acids can also be exchanged. The same is true for the diamines and polyalkyleneoxy-bis-(alkylenediamines). The invention is thus not limited to the polyamides cited as examples.

The amines, amides and polyamides cited under Component (D) can also be used as mixtures.

The suds suppressing dimethylpolysiloxanes cited under Component (E) are known commercial products, which are generally present as a mixture with binders such as pyrogenic silicic acid, emulsifiers and possibly solubilizing agents. Their proportion is preferably 8.2 to 8.8 wt% in reference to the laundry product used. The quantities are so adjusted that they prevent excessive suds production which would lead to overflow of suds from the wash liquor or to suppressing the mechanical movement of the wash load due to the formation of a "suds cushion", yet a certain amount of suds is present which indicates to the user, who probably associates washing with sudsing, that a washing process is going on.

The laundry product components cited under Component (F) are the usual additives, such as wash alkalis, bleaches, graying inhibitors, neutral salts, optical bleaches, enzymes, bactericides, brighteners, and coloring and perfuming agents.

Suitable wash alkalis are alkali metal silicates, especially sodium silicate with a ratio of Na2D : SiD2 = 1 : 0.5 to 1 : 3.5 and alkali carbonates, especially soda, and alkali metal borates. Bleaches are primarily active chlorine containing products such as sodium or lithium hypochlorite, di- or trichlor-isocyanuric acid or their alkali metal salts and chlorinated sulfonic acid amides (Chloramin T). Enzmes can also be used from the class of proteases, amylases and lipases, especially hapteness.

salts are especially sodium sulfate, biocides are halogenated diphenylmethane, salicylanilide and carbanilide.

laundry product can further contain optical bleaches, especially derivatives of diaminostilbene disulfonic acids or their salts. Suitable are, for example, salts of 4,4%-bis(2%aniline-4"-morpholine-1, 3, 5-triaziny1-6"-amino)-stilbene-2, 2"disulfonic acid or compounds of similar structure, which have in place of the morpholine group a diethanolamino group, a methylamino group or a beta-methoxy-ethylamino group. Also as. brighteners for polyamide fibers those of the type diarylpyrazoline are used, for example 1(-p-sulfonamidophenyl>-3- $(p-chlorophenyl)-\Delta^2-pyrazoline$  and compounds of similar structure, which in place of the sulfonamido group have a carboxymethyl or acetylamino group. Bubstituted aminocoumarins can also be used, e.g. 4-methyl-7-dimethylamino- or 4-methyl-7diethylamino coumarin. As polyamide brighteners the compounds 1-(2-benzimidazolyl)-2-(1-hydroxyethyl-2-benzimidazolyl)-ethylene and 1-ethyl-3-phenyl-7-diethylamino-carbostyril can also be used. As brighteners for polyester and polyamide fibers the compounds 2,5-di-(2-benzoxazolyl)-thiophene, 2-(2-benzoxazolyl)-naphtho-[2,3-b]-thiophene and 1,2-di-(5-methyl-2-benzoxazolyl)-ethylene are suitable. Brighteners of the substituted diphenylstyryl type can also be used. Also mixtures of the above brighteners can be used.

Buitable inhibitors of graying are especially carboxymethyl cellulose, methyl cellulose, as well as water soluble polyesters and polyamides of multivalent carboxylic acids and glycols or diamines which have free carboxyl groups, betaine groups or sulfobetaine groups which can form salts and colloidal water soluble polymers and copolymers of vinyl alcohols, vinylpyrrolidones, acrylamides and acrylonitriles.

The content of the above components in the laundry product can be as follows (in wt%):

- 1 to 20% wash alkalis
- 0.5 to 20% sodium sulfate
- 0.01 to 1% optical bleach
- 9 to 5% active oxygen or active chlorine
- 0.5 to 1% graying inhibitors
- 0 to 1% enzyme
- b to ix blocide, coloring and perfume agents

The good cleaning action which can be achieved by the washing process according to the invention is demonstrated in the following examples.

## Examples

A washing machine with a horizontally positioned, front loading drum having a load capacity of 4.5 kg dry wash was used. The drum turned at 1 revolution/sec. for 5 sec. in one direction and after

The machine was loaded with 4 kg of clean ballast (cotton) and 2 textile samples each measuring 20 x 10 cm. These samples were soiled under standard conditions (test soiling of the Laundry Research Institute, Krefeld) with iron oxide, kaolin, lampblack and skin fat and consisted of the following fiber material:

cotton (abbreviation BW)
cotton, finished (abbreviation BA)
polyester (abbreviation PE)
mixture of 50% polyester and 50% finished cotton
(abbreviation PB)

The laundry product had the following composition (wtx)

- 3.0 % tallow fatty alcohol, 14 ethoxy
- 0.5 % tallow fatty alcohol, 5 ethoxy
- 3.0 % tallow fatty acid soap (Na salt)
- 8.0 × n-dodecylbenzene sulfonate (Na salt)
- 68.8 % sodium tripolyphosphate
- 1.0 % Component D (see following explanation)
  - 0.5 % dimethylpolysiloxane suds suppressor (Component E)
  - 2.0 % soda
  - 2.5 % sodium silicate (NagD : SiDe = 1 : 3.3)
  - 0.2 % EDTA (Na salt)
  - 1.2 % Na carboxymethyl cellulose
  - 0.2 % biocide, coloring and perfume
  - 18.5 % sodium sulfate

Remainder water

The concentration used was 3 g/l, the wash liquor ratio was 1 : 3 and the hardness of the tap water used was 16 °CH. The washing test was done at 20°C and a wash time of 12, 20, 30 and 40 min., followed by three rinses, spinning, drying and then the reflectance of the samples was determined photometrically. The suds height was checked during the washing through the front window. There was no excess sudsing during any of the tests according to the invention. The suds reached the upper rim of the glass for only a short time; as a rule it reached only 2/3 to 3/4 of this height.

The following materials were used as Component D:

- Di) Reaction product of 1 mol of a straight chain internal  $\epsilon_{11}$   $\epsilon_{14}$  epoxide with 1 mol of diethanolamine, followed by ethoxylation with 1 mol of ethylene oxide (ED),
- DE> Coconut fatty acid monoethanolamide,
- D3) Dxazolidone derivative of linear, internal  $C_{14}$  epoxide, urea and N, N-dimethylpropylenediamine (see Formula 111),
- D4> Polyamide from epsilon-caprolactam (Ultramid B3>, reacted with ethylene oxide in a weight ratio of 1  $\pm$  4.45 ED,
- D5) Polyamide from adipic acid and hexamethylenediamine (U)tramid 6R) reacted with ethylene oxide in a weight ratio of 1 : 1.2 ED.

A wash time of 12 minutes, which is outside the required range

washing machines, did not give a completely satisfactory result {Table 1}.

In a second series of tests the tests were repeated in the absence of Components D and E (replaced by sodium sulfate). The results are shown in Table 2. Both the absence of Component D and the absence of the suds inhibitor (Component E) led to a decrease in the reflectance values. In addition a "conventional" i.e. a half hour wash treatment at  $60^{\circ}$ C was done. It was shown that the process according to the invention in some cases was superior, but at least was practically equal.

Table 1

| Example | Ď          | Textile        | * reflectance at a wash time of |              |                  |              |  |
|---------|------------|----------------|---------------------------------|--------------|------------------|--------------|--|
|         |            |                | 18                              | 20           | 38               | 40 min.      |  |
| 1       | D1         | ₽₩             | 47.4                            | 48.7         | 50.9             | 55.2         |  |
| 2       |            | <b>B</b> A     | 36. 3                           | 51.8         | 67.6             | 73. 1        |  |
| 3       |            | PE             | 49.2                            | <b>30.</b> 9 | 55.2             | 61.2         |  |
| 4       |            | PB             | 43.7                            | 45. 1        | 46.6             | 53. 1        |  |
| 5       | De         | <b>B</b> ₩     | 47.5                            | 49. 5        | 49. 5            | 51.4         |  |
| Б       |            | 2947           | 36.4                            | <b>59.</b> 2 | 65. 3            | <b>69.</b> 2 |  |
| 7       |            | PE             | 48. 4                           | 51.3         | 52. <del>8</del> | 57.8         |  |
| 8       |            | PB             | 42.5                            | 44.9         | 45.4             | 54. 2        |  |
| 9       | <b>D</b> 3 | ₽₩             | 47.7                            | 49. 9        | 53. 9            | 57.4         |  |
| 18      |            | <b>9</b> 6     | 56.5                            | 64. 9        | 71.1             | 74.4         |  |
| 22      |            | PE             | <b>5</b> e. 5                   | 36.6         | <b>69.</b> 6     | 64.2         |  |
| 12      |            | PB             | 45.7                            | 48. 9        | 51.1             | 57.9         |  |
| 13      | 24         | ₽₩             | 48. 8                           | 50. 1        | <b>5</b> 2. 1    | 54.6         |  |
| 24      |            | BA             | <b>58.</b> 2                    | 64. 1        | 78.3             | 74.4         |  |
| 15      |            | PE             | 46. 9                           | 49.4         | <b>53.</b> 8     | 57.4         |  |
| 16      |            | P <del>9</del> | 42.8                            | 44.8         | 45.5             | 50. 5        |  |
| 17      | 25         | <b>264</b>     | 47.8                            | 49.2         | 51.8             | 56.2         |  |
| 18      |            | BA             | 68. 9                           | <b>65.</b> 6 | 78. 9            | 74.5         |  |
| 19      |            | PE             | 47.7                            | 47.6         | 54.5             | 58. 8        |  |
| 20      |            | PÐ             | 43.3                            | 44.9         | 44.8             | 51.0         |  |
| Control | -          | 200            | <b>45.</b> 2                    | 45.8         | 47.1             | <b>50.</b> 6 |  |
|         |            | <b>BA</b>      | 53. 2                           | 59.7         | 63.9             | 65.7         |  |
|         |            | PE             | 43. 5                           | 44.6         | 46. 4            | 52.7         |  |
|         |            | PĐ             | 41.9                            | 41.9         | 42.3             | 49.7         |  |

Table 2

| Ехамріе | T <b>em</b> p<br>oC | Comp. | Comp.<br>E   | Wash time<br>min | Reflectance |               |               |
|---------|---------------------|-------|--------------|------------------|-------------|---------------|---------------|
|         |                     |       |              |                  | BA          | PE            | PB            |
| 21      | 200                 | 1× D2 | 9. 5×        | 48               | 73. 1       | 61.2          | 58. 1         |
| 55      | 200                 | 1× D3 | 9. 5x        | 48               | 74.4        | 64.2          | <b>5</b> 7. 9 |
| 23      | 200                 | 1× D4 | 9. 5×        | 48               | 74.4        | 57.4          | <b>30.</b> 6  |
| 24      | 200                 | 1× D5 | <b>8.</b> 5x | 40               | 74.5        | 58. 9         | 51.8          |
| Control | 200                 | _     | 9. 5×        | 48               | 65. 7       | <b>5</b> 2. 7 | 48.7          |
| Control | 200                 | -     |              | 48               | 59.6        | 49. 3         | 46.6          |
| Control | <b>59</b> 0         |       | _            | 30               | 75. 4       | 36.7          | 45. 2         |
|         |                     | • •   |              | linc. 15         |             |               |               |
|         |                     |       |              | min heating      | a> .        |               |               |

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